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Evaluation of Fused-Silica Capillary Columns for GC/ECD (Gas Chromatography with Electron Capture Detection) Analysis of Chlorinated Hydrocarbons Listed in EPA (Environmental Protection Agency) Method 8120 (Journal Version)

Acurex Corp., Mountain View, CA

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16. ABSTRACT

Four mega-bore, one wide-bore, and one narrow-bore fused-silica capillary columns were evaluated for their applicability to the GC/ECD analysis of 22 chlorinated hydrocarbons, some of which are currently targeted by EPA Method 8120. No one column can resolve all 22 compounds. Four compounds (two pairs) are coeluting on the SPB-35, DB-210, DB-WAX, and DB-519 fused-silica capillary columns, five compounds (two groups) are coeluting on the DB-1301 fused-silica capillary column, and ten compounds (five pairs) are coeluting on the SPB-5 fused-silica capillary column. The analysis time varies between 30 and 50 min. The order of elution of the chlorinated benzenes seems to depend on their boiling points rather than on the polarity of the liquid phase. The retention times of an additional nine chlorinated toluenes, eight chlorinated xylenes, and five chlorinated naphthalenes are also reported. Electron capture detector linearity is reported for the DB-210 fused-silica capillary column. Five brominated compounds were investigated as possible internal standards for Method 8120.

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Evaluation of Fused-Silica Capillary Columns for GC/ECD Analysis of Chlorinated Hydrocarbons Listed in EPA Method 8120

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Summary

Four mega-bore, one wide-bore, and one narrow-bore fused-silica capillary columns were evaluated for their applicability to the GC/ECD analysis of 22 chlorinated hydrocarbons, some of which are currently targeted by EPA Method 8120. No one column can resolve all 22 compounds investigated here. Four compounds (two pairs) are coeluting on the SPB-35, DB-210, DB-WAX, and DB-519 fused-silica capillary columns, five compounds (two groups) are coeluting on the DB-1301 fused-silica capillary column, and ten compounds (five pairs) are coeluting on the SPB-5 fused-silica capillary column. The analysis time varies between 30 and 50 min. The order of elution of the chlorinated benzenes seems to depend on their boiling points rather than on the polarity of the liquid phase. The retention times of an additional nine chlorinated toluenes, eight chlorinated xylenes, and five chlorinated naphthalenes are also reported. Electron capture detector linearity is reported for the DB-210 fused-silica capillary column. Five brominated compounds were investigated as possible internal standards for Method 8120.

1 Introduction

The Resource Conservation and Recovery Act (RCRA) of 1976 and its elements require the Environmental Protection Agency (EPA) to regulate the hazardous waste activities. Implementation and enforcement of RCRA requires analytical methodologies that will provide reliable data. The document "Test Methods for Evaluating Solid Waste", Office of Solid Waste Manual SW-846, which was revised recently [1], provides a compilation of methods for evaluating RCRA solid wastes for environmental and human hazards. One of the methods in this document addresses specifically the chlorinated hydrocarbons. Fifteen compounds, including seven chlorinated benzenes, three chlorinated toluenes, hexachloroethane, chloronaphthalene, hexachlorocyclohexane, hexachlorobutadiene, and hexachlorocyclopentadiene, are listed in Method 8120 (**Table 1**).

Table 1

Chlorinated hydrocarbons listed in the current EPA Method 8120.

Benzal chloride
Benzotrichloride
Benzyl chloride
2-Chloronaphthalene
1,2-Dichlorobenzene
1,3-Dichlorobenzene
1,4-Dichlorobenzene
Hexachlorobenzene
Hexachlorobutadiene
Hexachlorocyclohexane
Hexachlorocyclopentadiene
Hexachloroethane
Tetrachlorobenzenes
1,2,4-Trichlorobenzene
Pentachlorohexane[a]

[a] We believe that this should be pentachlorobenzene.

According to the current Method 8120, the determination of the 15 compounds is done by packed-column gas chromatography with electron capture detection (GC/ECD). The method specifies two gas chromatographic columns and five different column temperatures at which the gas chromatographic analyses can be performed [1]. Retention times, method detection limits, QC acceptance criteria, and accuracy and precision data are provided for only nine of the listed compounds. The separation of the tetrachlorobenzene and BHC isomers is not addressed, and 1,2,4-trichlorobenzene is listed as the only trichlorobenzene isomer. Furthermore, the use of one or more internal standards is specified in the analysis yet no compounds are suggested.

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Table 2

Gas chromatographic retention times for compounds investigated in this study and proposed for incorporation in the EPA Method 8120.

Compound no.	Compound	Retention time (min)[a]					
		SPB-5	SPB-35[b]	DB-210	DB-1301	DB-WAX	DB-519[c]
1	Benzal chloride	9.76	15.15	6.86	8.93	15.59	16.63
2	Benzotrichloride	12.36	17.72	7.85	10.81	15.10	19.56
3	Benzyl chloride	6.08	10.67	4.59	5.94	10.42	12.49
4	2-Chloronaphthalene	17.04	23.00	13.45	15.21	22.70	26.21
5	1,2-Dichlorobenzene	6.66	11.05	4.44	6.34	9.72	13.73
6	1,3-Dichlorobenzene	5.90	9.85	3.66	5.61	7.95	12.26
7	1,4-Dichlorobenzene	6.08	10.16	3.80	5.74	8.68	12.35
8	Hexachlorobenzene	26.21	32.48	19.23	23.31	27.44	37.33
9	Hexachlorobutadiene	12.36	16.53	5.77	10.05	10.08	17.07
10	α-BHC	25.96	33.06	22.21	24.17	36.86	35.79
11	β-BHC	27.56	35.41	25.54	27.78	47.46	41.50
12	γ-BHC	27.56	34.97	24.07	25.85	46.24	39.97
13	δ-BHC	28.89	37.11	26.16	29.00	31.33	43.69
14	Hexachlorocyclopentadiene	15.56	20.60	8.86	13.40	d)	21.37
15	Hexachloroethane	7.76	11.80	3.35	6.80	8.33	12.82
16	Pentachlorobenzene	21.15	27.08	14.86	18.53	22.70	28.99
17	1,2,3,4-Tetrachlorobenzene	17.04	22.91	11.90	15.01	20.33	24.98
18	1,2,4,5-Tetrachlorobenzene	15.82	21.01	10.18	13.40	17.28	23.29
19	1,2,3,5-Tetrachlorobenzene	15.56	20.93	10.18	13.40	17.00	23.29
20	1,2,4-Trichlorobenzene	10.90	15.96	6.86	9.58	13.57	18.44
21	1,2,3-Trichlorobenzene	12.00	17.43	8.14	10.78	15.62	19.97
22	1,3,5-Trichlorobenzene	9.52	14.00	5.45	8.24	10.42	16.76
	Total analysis time (min):	30	40	30	30	50	45
	Number of coeluting compounds:	10	4	4	5	4	4

a) The gas chromatographic conditions are given in Table 3.
b) 2-Chloronaphthalene and 1,2,3,4-tetrachlorobenzene are baseline resolved on a 30 m × 0.53 mm i.d. SPB-35 fused-silica capillary column with 0.5 μm film thickness [2].
c) This information was provided by *Roy Lautamo* of J & W Scientific.
d) Compound decomposes on column.

The purpose of this study was to evaluate several fused-silica capillary columns with chlorinated hydrocarbons and to establish gas chromatographic conditions for the GC/ECD analysis of the Method 8120 compounds. Twenty-two compounds listed in **Table 2** have been investigated. In addition, we determined the retention times for a series of chlorinated toluenes, xylenes, and naphthalenes that may interfere with the determinations of the 22 compounds. ECD linearity was also determined for one of the fused-silica capillary columns, and five brominated compounds were investigated as possible internal standards for Method 8120.

2 Experimental Section

2.1 Standards

High-purity benzyl chloride, 2-chloronaphthalene, 1,2-dichlorobenzene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, hexachlorobenzene hexachlorobutadiene, α-BHC, β-BHC, γ-BHC, δ-BHC, hexachlorocyclopentadiene, hexachloroethane, and 1,2,4-trichlorobenzene were obtained from the U.S Environmental Protection Agency Pesticides and Industrial Chemicals Repository. Benzal chloride, 1,2,3,5-tetrachlorobenzene, and the brominated compounds were obtained from Aldrich Chemical. The remainder of the compounds listed in Table 2 and the compounds listed in **Tables 4, 5,** and **6** were obtained from Ultrascientific Inc. Stock solutions of each test compound were prepared in isooctane (Burdick and Jackson) at concentrations of 1 mg/mL. Working calibration standards were prepared in isooctane by serial dilution of a composite stock solution prepared from the individual stock solutions.

2.2 Gas Chromatographic Procedures

Two Varian gas chromatographs equipped with a ^{63}Ni-electron capture detector were used throughout the course of this study. Six open tubular fused-silica capillary columns chemically bonded with different liquid phases were eva-

Table 3

GC operating conditions for the capillary column analyses.

	Column 1	Column 2	Column 3	Column 4	Column 5	Column 6[a]
Instrument	Varian 6000 equipped with ECD	Varian 3400 equipped with ECD	Varian 3400 equipped with ECD	Varian 3400 equipped with ECD	Varian 6000 equipped with ECD	FID
Column dimensions	15 m × 0.53 mm i.d.	30 m × 0.53 mm i.d.	30 m × 0.53 mm i.d.	30 m × 0.32 mm i.d.	30 m × 0.53 mm i.d.	30 m × 0.25 mm i.d.
Type of liquid phase	SPB-5 (methylphenyl silicone)	SPB-35 (35% phenyl methyl silicone)	DB-210 (trifluoropropyl methyl silicone)	DB-1301 (cyanopropyl methyl silicone)	DB-WAX (polyethylene glycol)	DB-519 (proprietary to J & W Scientific)
Film thickness (μm)	1.5	1.0	1.0	1.5	1.0	0.25
Carrier gas	Helium	Helium	Helium	Helium	Helium	Helium
Carrier flow (mL/min)	10	10	10	1.5	10	26.7 cm/s[b]
Temperature program	50°C to 175°C (hold 20 min) at 4°/min	50°C to 240°C (hold 10 min) at 4°/min	65°C to 175°C (hold 20 min) at 4°/min	100°C to 250°C (hold 20 min) at 5°/min	60°C to 170°C (hold 30 min) at 4°/min	60°C (hold 5 min) to 170°C (hold 20 min) at 5°/min
Injector temperature (°C)	220	220	220	220	200	250
Detector temperature (°C)	300	250	250	250	230	300
Injection volume (μL)	1.5	1.5	1.5	1.5	1.5	1.0
Type of injection	on-column	on-column	on-column	splitless (splitless time 60 sec; split flow 60 mL/min)	on-column	split (10:1)

[a] The analyses were performed by *Roy Lautamo* of J & W Scientific.
[b] Linear velocity.

Table 4

Retention times of various chlorinated toluenes analyzed on the fused-silica capillary columns evaluated in this study

	Retention time (min)[a]		
Compound	SPB-5	DB-210	DB-WAX
α,3-Dichlorotoluene	11.48	8.99	18.12
α,4-Dichlorotoluene	12.00	9.10	18.34
2,4,5-Trichlorotoluene	14.67	9.90	15.98
2,3,6-Trichlorotoluene	14.91	9.84	16.60
α,2,4-Trichlorotoluene	15.80	12.14	22.98
α,2,6-Trichlorotoluene	15.95	12.96	23.34
α,3,4-Trichlorotoluene	17.38	14.22	26.26
α,α,2,6-Tetrachlorotoluene	19.48	15.04	26.11
2,3,4,5,6-Pentachlorotoluene	24.94	18.96	27.66

[a] The gas chromatographic conditions are given in Table 3.

Table 5

Retention times of various chlorinated xylenes analyzed on the fused-silica capillary columns evaluated in this study.

	Retention time (min)[a]			
Compound	SPB-5	SPB-35	DB-210	DB-WAX
α-Chloro-o-xylene	8.89	14.31	12.88	12.58
α-Chloro-*m*-xylene	8.97	14.12	6.86	12.09
α,α'-Dichloro-o-xylene	15.36	21.98	12.94	25.09
α,α'-Dichloro-*m*-xylene	16.84	23.68	14.65	27.07
α,α'-Dichloro-*p*-xylene	16.96	23.93	14.86	27.26
α,α,α,α',α',α'-Hexachloro-*p*-xylene	26.73	34.42	19.87	31.41
α,α,α,α',α',α-Hexachloro-*m*-xylene	28.00	33.07	21.38	29.63
α,α',2,4,5,6-Hexachloro-*m*-xylene	34.81	41.95	29.57	32.94

[a] The gas chromatographic conditions are given in Table 3.

luated. The GC operating conditions are listed in **Table 3**. In each case, nitrogen was used as the makeup gas at a flow-rate of 40 mL/min. A Spectra Physics 4290 integrator was used to record the peak retention times and to integrate the peak areas.

Table 6

Retention times of various chlorinated naphthalenes analyzed on the fused-silica capillary columns evaluated in this study.

	Retention time (min)[a]			
Compound	SPB-5	SPB-35	DB-210	DB-WAX
1,4-Dichloronaphthalene	22.17	28.61	17.43	26.33
2,7-Dichloronaphthalene	22.36	28.82	18.29	26.28
1,5-Dichloronaphthalene	22.37	28.81	17.62	26.32
1,2-Dichloronaphthalene	22.86	29.37	18.62	26.28
1,2,3,4-Tetrachloro-naphthalene	31.97	39.37	26.32	39.84

[a] The gas chromatographic conditions are given in Table 3.

3 Results and Discussion

GC/ECD chromatograms of a composite solution containing the 22 compounds analyzed on five of the six fused-silica capillary columns used in this study are shown in **Figures 1** through **5**. Table 2 summarizes the retention times of the 22 test compounds for each fused-silica capillary column. No one column can resolve all 22 compounds. Under the GC operating conditions that are given in **Table 3** the number of compounds not resolved on the six columns is as follows: ten compounds (five pairs) on the SPB-5; four compounds (two pairs) on the SPB-35, DB-210, DB-WAX, and DB-519; and five compounds (two groups) on the DB-1301.

The only fused-silica capillary column that can resolve all of the chlorinated benzene isomers is the DB-WAX. It is interesting to note that on all columns tested the elution order of all the polychlorinated benzene isomers is identical regardless of the liquid phase of the column. The order is 1,3-, 1,4-,

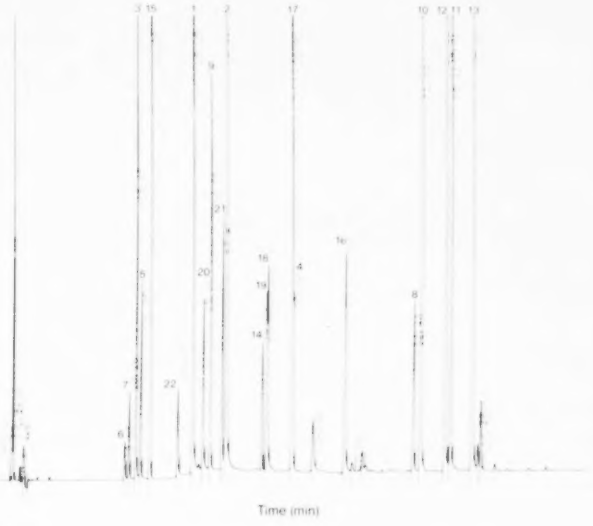


Figure 2

GC/ECD chromatogram of the Method 8120 compounds analyzed on an SPB-35 fused-silica capillary column; the GC operating conditions are given in Table 3. For peak identification refer to Table 2. Standards in isooctane at concentrations 5 times as high as those given in Table 7.

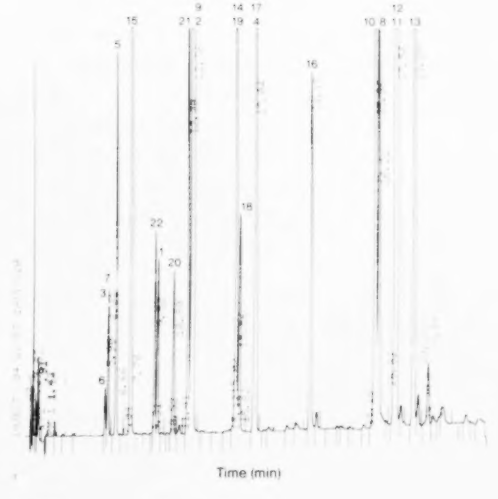


Figure 1

GC/ECD chromatogram of the Method 8120 compounds analyzed on an SPB-5 fused-silica capillary column; the GC operating conditions are given in Table 3. For peak identification refer to Table 2. Standards in isooctane at concentrations 5 times as high as those given in Table 7.

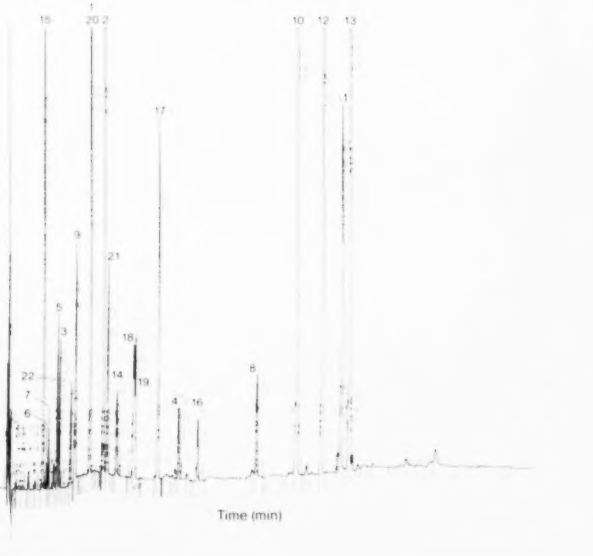


Figure 3

GC/ECD chromatogram of the Method 8120 compounds analyzed on a DB-210 fused-silica capillary column; the GC operating conditions are given in Table 3. For peak identification refer to Table 2. Standards in isooctane at concentrations listed in Table 7.

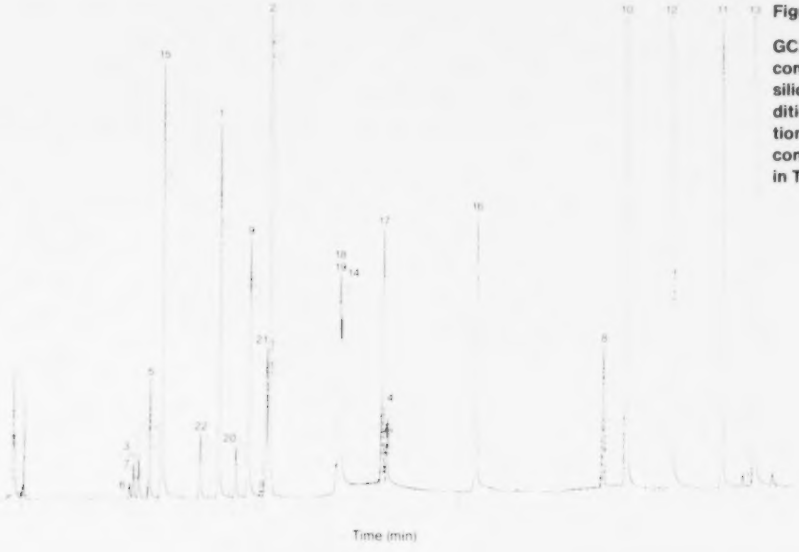


Figure 4

GC/ECD chromatogram of the Method 8120 compounds analyzed on a DB-1301 fused-silica capillary column; the GC operating conditions are given in Table 3. For peak identification refer to Table 2. Standards in isooctane at concentrations 10 times as high as those given in Table 7.

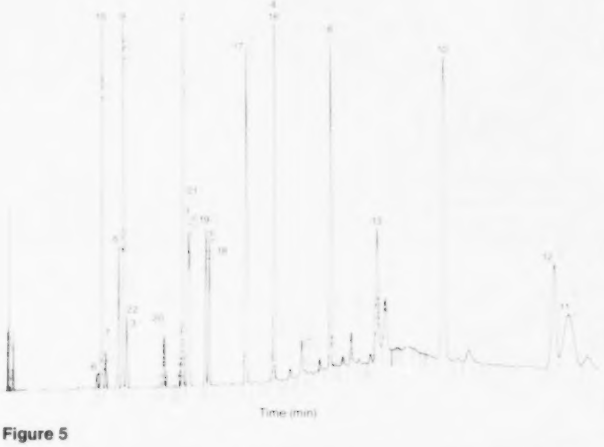


Figure 5

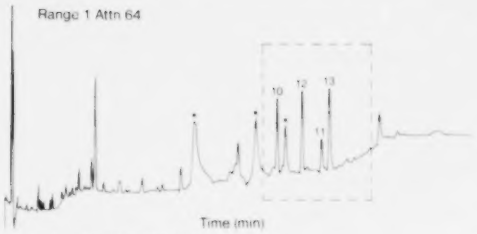
GC/ECD chromatogram of the Method 8120 compounds analyzed on a DB-WAX fused-silica capillary column; the GC operating conditions are given in Table 3. For peak identification refer to Table 2. Standards in isooctane at concentrations 10 times as high as those given in Table 7.

1,2-, 1,3,5-, 1,2,4-, 1,2,3-, 1,2,3,5-, 1,2,4,5-, and 1,2,3,4-chlorinated benzene. The same is true for the elution order of the dichlorobenzenes in the packed columns specified in Method 8120. Thus, it appears that the elution order of these compounds is dictated by the order of their boiling points. Another interesting observation is the fact that the DB-WAX column properties differ slightly from those of the Supelcowax 10 column, despite equivalency claims by the DB-WAX column manufacturer. To our surprise, we were not able to elute the BHCs from a 30 m × 0.53 mm i.d. Supelcowax 10 fused-silica capillary column (1 μm film thickness). The elution order of the other chlorinated compounds on the DB-WAX and Supelcowax 10 columns is identical.

The coeluting compounds and the respective columns are:

- SPB-5: Benzyl chloride/1,4-dichlorobenzene
  Benzotrichloride/hexachlorobutadiene
  2-Chloronaphthalene/1,2,3,4-tetrachlorobenzene
  β-BHC/γ-BHC
  Hexachlorocyclopentadiene/1,2,3,5-tetrachlorobenzene
- SPB-35: 2-Chloronaphthalene/1,2,3,4-tetrachlorobenzene
  1,2,3,5-/1,2,4,5-tetrachlorobenzene
- DB-210: Benzal chloride/1,2,4-trichlorobenzene
  1,2,3,5-/1,2,4,5-tetrachlorobenzene
- DB-1301: Benzotrichloride/1,2,3-trichlorobenzene
  Hexachlorocyclopentadiene/1,2,3,5-/1,2,4,5-tetrachlorobenzene
- DB-WAX: Benzal chloride/1,3,5-trichlorobenzene
  2-Chloronaphthalene/pentachlorobenzene
- DB-519: 1,3-/1,4-Dichlorobenzene
  1,2,3,5-/1,2,4,5-tetrachlorobenzene

The retention times of several chlorinated toluenes, xylenes, and naphthalenes are presented in Tables 4, 5, and 6, respectively. As in the case of the chlorinated benzene isomers, these compounds also seem to follow the same elution pattern regardless of the liquid phase of the column. There are some exceptions, however, and not all compounds reported in Tables 4, 5, and 6 are completely resolved on the fused-silica columns investigated. Chlorinated naphthalenes with two chlorines were the most difficult compounds to separate. In fact, the DB-WAX cannot separate them at all, and the SPB-5 and SPB-35 cannot separate 2,7- and 1,5-dichloronaphthalene.



*Present in isooctane blank

Figure 6

GC/ECD chromatograms of Method 8120 compounds (concentration 0.0002 to 0.1 ng/μL) analyzed on a DB-210 fused-silica capillary column; GC operating conditions are given in Table 3. For identification of peaks 10, 11, 12, and 13, refer to Table 2.

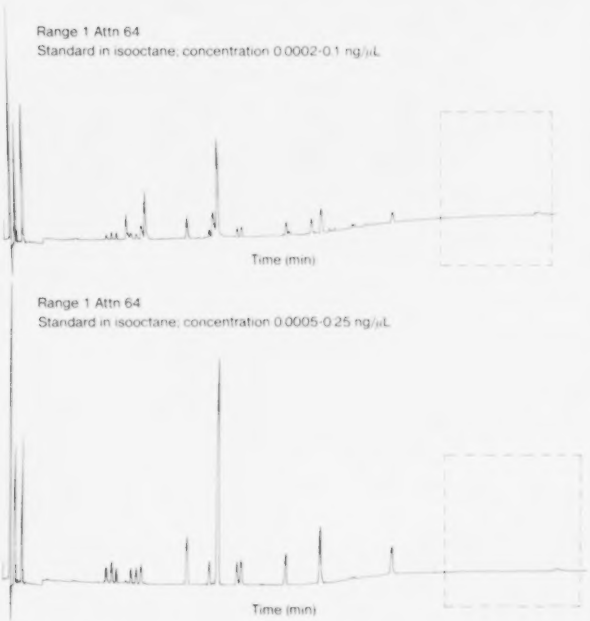

Range 1 Attn 64
Standard in isooctane; concentration 0.0002-0.1 ng/μL
Time (min)
Range 1 Attn 64
Standard in isooctane; concentration 0.0005-0.25 ng/μL
Time (min)

Figure 7

GC/ECD chromotograms of Method 8120 compounds at two different concentrations analyzed on a DB-WAX fused-silica capillary column. GC operating conditions are given in Table 3. Area marked in interrupted line represents the elution window for BHCs.

The DB-210 fused-silica capillary column resolved more compounds than any of the other columns tested and is therefore proposed for incorporation into EPA Method 8120. The DB-WAX fused-silica column is being considered for confirmatory purposes since this column resolves the tetrachlorobenzene isomers; however, additional column evaluation studies are in progress to establish the elution order of the 22 chlorinated hydrocarbons from various polyethylene glycol bonded fused-silica capillary columns that are commercially available. Except for the BHC isomers, the instrument detection limits determined with the DB-210 and DB-WAX fused-silica capillary columns are comparable. The BHCs, if present below 0.1 ng per injection can only be detected with the DB-210 fused-silica capillary column. **Figure 6** shows a GC/ECD chromatogram of a calibration solution containing BHCs at 0.002 ng/μL analyzed on the DB-210 column. When the same calibration solution was injected on the DB-WAX fused-silica capillary column, all Method 8120 compounds, except BHCs, were found. By increasing the concentration of the Method 8120 compounds, we were still not able to detect the BHCs (**Figure 7**).

The calibration factors of the 22 compounds were determined at two-week interval on three separate days using GC/ECD and the DB-210 fused silica capillary column. Each time, a seven-point multilevel calibration was performed. The values of the calibration factors (**Table 7**), although very reproducible for the three sets of data, vary widely from compound to compound. The variation in detector response is not only the result of the increased number of halogens because we found large differences even among isomers with the same number of halogens (*e. g.* the average calibration factors for 1,3-dichlorobenzene and 1,2-dichlorobenzene for day 1 are 9.6×10^3 and 8.8×10^4, respectively).

The ECD is least sensitive to the dichlorobenzenes and to 2-chloronaphthalene; for this reason, the concentrations of these compounds in the calibration solutions are 10 to 200 times as high as the concentrations of the other compounds.

The detector response was linear for all compounds over a 10-fold concentration range when using the DB-210 fused-silica capillary column. The linear regression coefficients were all greater than 0.95. The minimum detectable levels vary from 0.1 pg for hexachloroethane to 500 pg for the dichlorobenzenes and for 2-chloronaphthalene.

The five brominated compounds listed in **Table 8** were investigated as possible internal standards for Method 8120. Two of these compounds, *m*-dibromobenzene and 1,2,4,5-tetrabromobenzene, coelute with some of the chlorinated hydrocarbons; therefore, they cannot be used as internal standards. Test results with 1,3,5-tribromobenzene, spiked into clean hexane at a nominal concentration of 1 ng/μL, indicated excellent precision. The coefficients of variation for ten consecutive injections were 0.067 percent for retention time and 0.8 percent for calibration factors.

To establish method accuracy, we have obtained two EPA check samples and analyzed them on the DB-210 fused-silica capillary column. The results are presented in **Table 9**. Despite the somewhat high biases for three of the compounds, we found that these results are better than those currently reported by EPA for packed-column GC/ECD analysis. It is obvious that modification of the current EPA Method 8120 is required to improve the gas chromato-

Table 7

Multilevel calibration data for the test compounds analyzed on the DB-210 fused-silica capillary column.

Compound[a]	Concentration[b] (ng/μL)	Average CF[b/c]			Linear regression coefficient[b]		
		Day 1	Day 14	Day 26	Day 1	Day 14	Day 26
Hexachloroethane	0.01	26.5×10^6	26.5×10^6	30.7×10^6	0.9763	0.9715	0.9785
1,3-Dichlorobenzene	1.0	9.6×10^3	10.1×10^3	10.9×10^3	0.9937	0.9964	0.9894
1,4-Dichlorobenzene	1.0	2.5×10^4	2.7×10^4	3.0×10^4	0.9884	0.9916	0.9875
1,2-Dichlorobenzene	1.0	8.8×10^4	9.2×10^4	10.6×10^4	0.9992	0.9940	0.9975
Benzyl chloride	0.1	7.9×10^4	8.2×10^4	9.8×10^4	0.9981	0.9986	0.9964
1,3,5-Trichlorobenzene	0.1	4.7×10^5	6.0×10^5	5.5×10^5	0.9921	0.9953	0.9881
Hexachlorobutadiene	0.01	14.2×10^6	17.6×10^6	17.2×10^6	0.9995	0.9958	0.9991
Benzal chloride	0.1	15.2×10^5	16.9×10^5	17.8×10^5	0.9736	0.9630	0.9791
1,2,4-Trichlorobenzene	0.1	15.2×10^5	16.9×10^5	17.8×10^5	0.9736	0.9630	0.9791
Benzotrichloride	0.1	6.9×10^6	7.4×10^6	7.9×10^6	0.9523	0.9464	0.9717
1,2,3-Trichlorobenzene	0.1	12.7×10^5	16.3×10^5	15.6×10^5	0.9934	0.9962	0.9966
Hexachlorocyclopentadiene	0.01	6.3×10^6	7.5×10^6	8.5×10^6	0.9868	0.9932	0.9812
1,2,4,5-Tetrachlorobenzene	0.1	7.2×10^5	10.5×10^5	8.9×10^5	0.9976	0.9958	0.9905
1,2,3,5-Tetrachlorobenzene	0.1	7.7×10^5	10.9×10^5	8.9×10^5	0.9976	0.9958	0.9905
1,2,3,4-Tetrachlorobenzene	0.1	20.9×10^5	26.7×10^5	24.4×10^5	0.9967	0.9851	0.9966
2-Chloronaphthalene	2.0	2.5×10^4	2.9×10^4	2.8×10^4	0.9952	0.9967	0.9899
Pentachlorobenzene	0.01	4.2×10^6	4.5×10^6	5.1×10^6	0.9963	0.9952	0.9968
Hexachlorobenzene	0.01	8.9×10^6	10.4×10^6	12.2×10^6	0.9846	0.9730	0.9732
α-BHC	0.1	7.3×10^6	7.4×10^6	8.6×10^6	0.9619	0.9647	0.9765
γ-BHC	0.1	9.5×10^6	8.6×10^6	10.7×10^6	0.9600	0.9595	0.9689
β-BHC	0.1	4.1×10^6	3.8×10^6	5.3×10^6	0.9777	0.9871	0.9904
δ-BHC	0.1	9.1×10^6	9.7×10^6	10.0×10^6	0.9615	0.9622	0.9820

[a] The compounds were arranged in the order in which they elute from the DB-210 fused-silica capillary column.

[b] The number of data points is 7; the concentration levels were 1X; 2X; 4X; 5X; 7X; 8X; 10X those listed.

[c] The calibration factor (CF) is the ratio of instrument response to the amount injected in ng.

Table 8

Brominated compounds investigated as possible internal standards for Method 8120

Compound	Retention time (min)[a]			Comments
	SPB-35	DB-210	DB-1301	
m-Dibromobenzene	16.58	6.88	9.99	Coelutes with hexachlorobutadiene on SPB-35, with benzal chloride and 1,2,4-trichlorobenzene on DB-210, and with hexachlorobutadiene on DB-1301
2,5-Dibromotoluene	20.36	9.55	12.94	
1,3,5-Tribromobenzene	24.38	11.68	16.03	
α,α'-Dibromo-*m*-xylene	29.45	18.43	20.22	
1,2,4,5-Tetrabromobenzene	33.87	19.20	24.14	Coelutes with hexachlorobenzene on DB-210 and with α-BHC on DB-1301

[a] The gas chromatographic conditions are given in Table 3.

graphic analysis of the chlorinated hydrocarbons; this work is being performed at the Acurex Corporation laboratory.

Selection of the DB-210 fused-silica capillary column as a primary column may be surprising to the reader. The DB-210 is a trifluoropropyl methyl silicone liquid phase and column bleed could result in a high background because of fluorine. However, it has been our experience that by maintaining the detector temperature to 250°C and by proper column conditioning prior to use, the detector contamination is minimized and the ECD detector system will not require any baking during routine operation (provided the

Table 9

Method accuracy established by analysis of EPA check samples WP-685 and WP-186[a,b].

Compound	True value (μg/mL)	Value found (μg/mL)	Bias (percent)
EPA Check sample WP-685			
α-BHC	100	117	+17
γ-BHC	100	85	−15
EPA Check sample WP-186			
1,3,5-Trichlorobenzene	100	107	+ 7
1,2,3-Trichlorobenzene	100	85	−15
1,2,4,5-Tetrachlorobenzene	100	105	+ 5
1,2,3,4-Tetrachlorobenzene	100	96	− 4

[a] The analyses were performed using the DB-210 fused-silica capillary column and the GC operating conditions listed in Table 2.

[b] The current EPA Method 8120 does not give any information regarding method accuracy for these compounds. Values given for the recovery of 1,2,4-trichlorobenzene from water is 20 to 134 percent [1].

samples are relatively clean and the concentrations of the test compounds are within the linear range of the detector).

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[1] Test Methods for Evaluating Solid Waste, Vol 1B: Laboratory Manual Physical/Chemical Methods, SW-846, 3rd Ed., U.S. Environmental Protection Agency, Washington, D.C., November 1986.

[2] *N. Mosesman*, personal communication, 1987.

Notice

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